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Europäisches Patentamt
European Patent Office
Office européen des brevets

(11) Publication number:

0 339 792
A2

(12)

EUROPEAN PATENT APPLICATION

(21) Application number: 89302966.0

(51) Int. Cl.4: **B29C 33/60 , C08K 5/09 ,**
C08K 5/29 , //B29K75:00

(22) Date of filing: 23.03.89

(30) Priority: 31.03.88 GB 8807684
14.06.88 GB 8814024

(43) Date of publication of application:
02.11.89 Bulletin 89/44

(84) Designated Contracting States:
AT BE CH DE ES FR GB IT LI NL SE

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(54) Internal mould release composition.

(57) An internal mould release composition, suitable for use in the production of moulded articles by the RIM process, comprising :

- a) a metal salt of an organic acid, said metal salt having mould release properties, and
- b) a compatibilising amount of a guanidine or isourea compound of molecular weight below 1500.

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Internal mould release compositions

This invention relates to internal mould release compositions and to their use in the production of moulded articles by the reaction injection moulding process from reaction mixtures containing organic polyisocyanates and isocyanate-reactive components.

The production of moulded articles, for example automotive body parts, from organic polyisocyanates and isocyanate-reactive components by the reaction injection moulding (RIM) process is well established. In the process, two highly reactive liquid streams, one containing a polyisocyanate and the other containing isocyanate-reactive components, for example polyol and/or polyamine reactants, are impingement mixed and rapidly injected into a mould cavity. By this method, high production rates of moulded polyurethane, polyurea and related polymeric articles having excellent physical properties can be achieved.

Whilst the RIM process is eminently suited to the mass production of isocyanate-based moulded articles, it is essential to its successful and economic operation that the moulded product can be easily removed from the mould without being damaged. One method of facilitating demoulding is to coat the internal surfaces of the mould with a mould release agent, for example a wax or soap. This procedure has the disadvantage that application of the release agent has to be repeated almost every time the mould is used, adding substantially to the time and cost of the process.

In order to overcome the problems associated with external mould release agent, it has been proposed to use release agents of the internal type pre-blended with either the polyisocyanate or the isocyanate-reactive component. One type of internal release agent which has been successfully used in the production of other moulded plastics articles comprises metal carboxylates such as zinc stearate.

Unfortunately, such salts are soluble in neither the polyisocyanate nor with most of the usual isocyanate-reactive components. It has been proposed, therefore, to use the metal salts in conjunction with certain materials which have the effect of compatibilising the salts with the isocyanate-reactive compounds. Thus, for example, it has been proposed in European Patent Publications 119471, 173888 and 190317 to employ various amino compounds as compatibilisers.

It has now been found out that guanidine and isourea containing compounds show superior performances to those of the amino compounds proposed in the prior art, when used with metal salts of organic acids and isocyanate-reactive compounds in the manufacture of moulded polyurethane, polyurea and like polymers by processes such as the RIM-process.

Accordingly, the invention provides an internal mould release composition comprising :

- (a) a metal salt of an organic acid, said metal salt having mould release properties, and
- (b) a compatibilising amount of a guanidine or isourea compound.

The term "compatibiliser", as used herein, refers to the formation of a stable dispersion, colloidal suspension and/or solution of the metal salt (having internal mould release properties) in the isocyanate reactive (resin) component of the reaction injection moulding (RIM) system.

The compatibilising agents of the invention have the advantage of improved stability (to precipitation of the metallic salt internal mold release agent) and better control over reactivity (i.e. flow, gel time).

The expression "a compatibilising amount" used herein means an amount of the guanidine or isourea compound sufficient to provide a substantially stable solution or dispersion of mould release composition in an isocyanate-reactive compound or compounds. Clearly, this amount will vary depending on the nature of the metal salt, the nature of the isocyanate-reactive compound and their relative proportions as well as on the specific structure of the guanidine or isourea.

The metal salts present in the compositions of the invention may be any metal salts which are derived from organic acids and which have mould release properties. Such salts have been fully described in the prior art relating to the moulding of polyurethanes, polyureas and other resins.

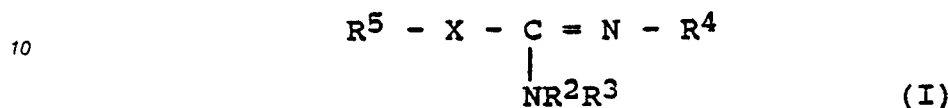
The organic acids from which the salts may be derived particularly include carboxylic acids containing from 8 to 24 carbon atoms. Such acids may be aromatic or cycloaliphatic but are preferably aliphatic monocarboxylic acids which may be straight-chain or branched, saturated or unsaturated and may carry substituents such as hydroxy, amino or amido groups. The most preferred aliphatic monocarboxylic acids are the so-called "fatty acids" having from 10 to 18 carbon atoms, for example lauric, palmitic, stearic, isostearic, oleic, linoleic and ricinoleic acids and mixtures of any of these.

The metal salts may be salts of any metal of Groups Ia, Ib, IIa and IIb of the Periodic Table or of other metals such as chromium, molybdenum, iron, cobalt, nickel, aluminium, tin, lead, antimony or bismuth. The preferred metals are lithium, magnesium, calcium, barium, iron, cobalt, nickel, copper, zinc, cadmium and aluminium and mixtures thereof.

As examples of particularly suitable salts there may be mentioned zinc stearate, zinc oleate, zinc palmitate, zinc laurate, zinc octoate, zinc behenate, zinc ricinoleate and the calcium, magnesium, nickel and copper salts of lauric, palmitic, stearic and oleic acids. The most preferred salt is zinc stearate.

The guanidine and isourea compounds used as compatibilisers in the compositions of the invention are preferably capable of reacting with isocyanates, without the liberation of monomeric by-products.

Suitable guanidine or isourea compounds are represented by formula (I) :



wherein X represents O, S or NR¹ and

wherein each of R¹ to R⁵ independently or together represent, H or an organic radical.

It is noted that in the above formula at least two of the three substituents attached to the imino-units can be joined together to form at least one non-aromatic ring, preferably a 5 or 6 membered ring.

Suitable organic radicals include :

a) alkyl radicals, preferably of 1 to 10 C atoms, more preferably 1 to 6 C atoms;

b) cycloalkyl radicals, preferably of 5 to 7 C atoms;

c) aralkyl and aryl radicals of 6 to 18 C atoms - preferably substituted with electron donating substituents such as hydroxy, alkoxy, N,N-dialkyl-amino etc., such as benzyl, phenyl and phenylethyl;

d) polymeric chains containing hetero atoms (such as O, N or S) which may be selected from polyether chain, polyester chains, polyacetals, polycarbonates, etc.

Optionally, each of R¹ to R⁵, when not H, may be further substituted with an additional guanidine or isourea group or residue obtained by the removal of anyone of R¹ to R⁵ radicals from above formula (I) compound.

Preferred guanidine or isourea compounds for use in the compositions of the invention contain from 1 to 4 such groups.

It is preferred that the guanidine and isourea compounds have molecular weights not greater than 1500, preferably in the range 60 to 600, for example 100 to 600.

The preparation of guanidines or isoureas in both cyclic and acyclic forms is well known in the literature.

Isoureas are generally prepared by the reaction of an alcohol with a carbodiimide in the presence of a suitable catalyst. The alcohol component may be aliphatic, as described in E. Schmidt, F. Moosmuller, Lieb. Ann. 597, 235, (1956), or aromatic as in E. Vowinkel, Chem. Ber., 96, 1702, (1963). The catalyst employed in these reactions are frequently chloride salts of copper, such as the use of copper (I) chloride in E. Vowinkel, I. Buthe, Chem. Ber., 107, 1353, (1974), or copper (II) chloride, as in E. Schmidt, E. Dabritz, K. Thulke, Lieb. Ann., 685, 161, (1965).

However the reaction can also be carried out by the addition of an alkali metal to the alcohol component as exemplified by the use of sodium metal in H.G. Khorana, Canad. J. Chem. 32, 261, 1953.

Guanidines can be prepared by the reaction of an amine with a carbodiimide in a manner similar to that outlined in the references cited above. Alternatively alkylguanidines may be prepared by the reaction of an alkylamine salt with dicyandiamide as in E.A. Werner, J. Bell, J. Chem. Soc., 121, 1790, (1922). In yet another method s-methylthiourea sulphate is combined with an alkylamine as described in "Heterocyclic Chemistry", A. Albert, Althone Press, London, 1968.

Examples of especially suitable guanidine and isoureas for use in the compositions of the invention include reaction products of carbodiimides such as dicyclohexyl carbodiimide, dimethylcarbodiimide, diphenylcarbodiimide with amines such as ammonia cyclohexylamine, methylamine, ethylamine, isopropylamine, etc. or alcohols such as methanol, ethanol, n-propanol, isopropanol, n-butanol, t-butanol, isobutanol, pentanol, hexanol, heptanol, phenol, ethoxyethyl ethanol, 1-methoxy-ethanol, and the like, or diethanolamine and triethanolamine.

Typical guanidine compounds include : tricyclohexylguanidine, tetramethylguanidine, diphenylguanidine, N,N'-ditolylguanidine, (dichlorophenyl)-guanidine, (4-chloro-2-methylphenyl)-guanidine, (4-chlorophenyl)-guanidine, N,N'-dixylguanidine (3-chlorophenyl)-guanidine.

Typical isourea compounds include : N,N'-dicyclohexyl-O-ethylisourea.

Mixtures of guanidines and/or isoureas may be present in the compositions if desired.

The compositions of the invention typically contain from about 0.1 to about 40 % preferably from about 1 to about 20 % of metal salt on a weight basis.

The compositions may be conveniently prepared by agitating one or more metal salts as hereinbefore
5 defined with one or more guanidine and/or isourea compounds at ambient or elevated temperatures until a solution or stable dispersion is obtained. In some cases, stirring for several hours at temperatures of up to 80° C or even higher may be necessary.

As indicated above, the internal mould release compositions of the invention are useful in the
10 production of moulded articles by a process comprising reacting in a suitable mould a polyisocyanate composition with a isocyanate-reactive composition comprising one or more compounds containing a plurality of isocyanate-reactive groups in the presence of said mould release composition.

In order to facilitate the incorporation of the internal mould release composition of the invention into the reaction mixture used in the moulding process, it is usually desirable to pre-blend said composition into at least part of the isocyanate-reactive composition employed in the process.

15 Accordingly, in another aspect of the invention, there is provided an isocyanate-reactive composition comprising at least one compound having a plurality of isocyanate-reactive groups having dissolved or dispersed therein an effective amount of an internal mould release composition comprising :

(a) a metal salt of an organic acid, said metal salt having mould release properties, and

(b) a compatibilising amount of a guanidine or isourea compound,

20 the metal salt being incompatible with the compound having a plurality of isocyanate-reactive groups in the absence of the compatibiliser, under the conditions used for preparing and processing said isocyanate reactive composition.

Said isocyanate-reactive composition may be prepared by combining all the ingredients in a single operation (stirring and heating as necessary to provide complete compatibilisation) or, if desired, by pre-
25 blending any two or more of the individual ingredients.

The expression "an effective amount" used herein means an amount of the mould release composition of the invention sufficient to provide improved mould release when the isocyanate-reactive composition is used in a moulding process. Where the isocyanate-reactive composition contains more than one isocyanate-reactive compound, the requirement of incompatibility with the metal salt means that the metal
30 salt is incompatible with at least one of the isocyanate-reactive compounds present in the composition, under the conditions used for preparing and processing said composition.

Compounds containing a plurality of isocyanate-reactive groups which may be present in the isocyanate-reactive compositions of the invention include compounds having molecular weights of from about 60 to about 12000, containing an average of two or more isocyanate-reactive groups per molecule.
35 Examples of isocyanate-reactive groups which may be present in these compounds include hydroxyl, thiol, primary amino, secondary amino, imino-functional and enamine groups, and mixtures of such groups.

Particularly important isocyanate-reactive compositions for use in RIM processes contain a combination of a relatively high molecular weight polymer containing a plurality of isocyanate-reactive groups (usually referred to as a "softblock") and a relatively low molecular weight compound containing a plurality of
40 isocyanate-reactive groups, usually known as a chain extender.

Thus, in a further aspect of the invention, there is provided an isocyanate-reactive composition comprising :

(i) at least one polymer containing a plurality of isocyanate-reactive groups and having a number average molecular weight of from 1500 to about 12000;

45 (ii) at least one chain extender having a molecular weight less than 1500, and

(iii) an effective amount of an internal mould release composition comprising :

(a) a metal salt of an organic acid, said metal salt having mould release properties, and

(b) a compatibilising amount of a guanidine or isourea compound,

50 the metal salt being incompatible with the combination of (i) and (ii) in the absence of the compatibiliser, under the conditions used for preparing and/or processing said isocyanate reactive composition.

Polymers containing a plurality of isocyanate-reactive groups which may be present in the isocyanate-reactive compositions of the invention include polyols, polyamines, imino-functional polymers, enamine
55 containing polymers and mixtures thereof.

Polymeric polyols having molecular weights in the range from 1500 to about 12000 are well known as polyurethane components and may be prepared by methods fully described in the prior art. As examples of suitable polyols there may be mentioned polythioethers, polyesters, polyesteramides, polycarbonates,

polyacetals, polyolefins, polysiloxanes and, especially, polyethers.

Polyether polyols which may be used include products obtained by the polymerisation of a cyclic oxide, for example ethylene oxide, propylene oxide or tetrahydrofuran in the presence, where necessary, of polyfunctional initiators. Suitable initiator compounds contain a plurality of active hydrogen atoms and
 5 include water, polyols, for example ethylene glycol, propylene glycol, diethylene glycol, resorcinol, bisphenol A, glycerol, trimethylolpropane, 1,2,6-hexanetriol, triethanolamine, pentaerythritol, sorbitol or sucrose, ammonia, primary monoamines, for example aniline or benzylamine, polyamines, for example ethylene diamine, hexamethylene diamine, toluene diamines, diaminodiphenylmethanes and polymethylene
 10 polyphenylene polyamines obtained by the condensation of aniline and formaldehyde, and aminoalcohols, for example ethanolamine and diethanolamine. Mixtures of initiators and/or cyclic oxides may be used.

Especially useful polyether polyols include polyoxypropylene and poly(oxyethylene-oxypropylene) diols and triols obtained by the simultaneous or sequential addition of ethylene and propylene oxides to appropriate di- or trifunctional initiators as fully described in the prior art. Mixtures of the said diols and triols are often particularly useful.

15 Because of their enhanced reactivity, ethylene oxide tipped polyols are often preferred.

Polyester polyols which may be used include hydroxyl terminated reaction products of polyhydric alcohols such as ethylene glycol, propylene glycol, diethylene glycol, 1,4-butanediol, polyether polyols, bis-(hydroxyethyl) terephthalate, glycerol, trimethylolpropane or pentaerythritol or mixtures thereof with polycarboxylic acids, especially dicarboxylic acids or their ester-forming derivatives, for example succinic, glutaric
 20 and adipic acids or their dimethyl esters, sebacic acid, phthalic anhydride, tetrachlorophthalic anhydride or dimethyl terephthalate. Polyesters obtained by the polymerisation of lactones, for example caprolactone, in conjunction with a polyol, may also be used. Polyesteramides may be obtained by the inclusion of aminoalcohols such as ethanolamine in polyesterification mixtures.

Polythioether polyols which may be used include products obtained by condensing thiodiglycol (either
 25 alone or with other glycols), with dicarboxylic acids, formaldehyde, alkylene oxides, aminocarboxylic acids, or combinations thereof.

Polycarbonate polyols which may be used include products obtained by reacting diols such as 1,3-propanediol, 1,4-butanediol, 1,6-hexanediol, diethylene glycol or tetraethylene glycol with diaryl carbonates, (for example diphenyl carbonate), with cyclic carbonates (i.e. ethylene carbonate), or with phosgene.

30 Polyacetal polyols which may be used include those prepared by reacting glycols such as diethylene glycol, triethylene glycol or hexanediol with formaldehyde. Suitable polyacetals may also be prepared by polymerising cyclic acetals.

Suitable polyolefin polyols include hydroxy-terminated homo- and copolymers and suitable polysiloxane polyols include polydimethylsiloxane diols and triols, wherein the hydroxyl groups are organically bound.

35 Polymeric polyamines having molecular weights in the range from 1500 to about 12000 are well known as components of polyurea forming reaction mixtures and may be prepared by methods fully described in the prior art. As examples of suitable polyamines, there may be mentioned amino-terminated polythioethers, polyesters, polyesteramides, polycarbonates, polyacetals, polyolefins, polysiloxane and, especially, polyethers obtained by replacing the hydroxy groups of the corresponding polyols partially or completely
 40 by secondary or preferably primary amino groups. The preferred polymeric polyamines are polyether diamines and triamines, especially polyoxypropylene diamines and triamines.

Imino-functional polymers having molecular weights in the range from 1500 to about 12000 have been described in US Patent No. 4,794,129 together with methods for their preparation. The preferred imino-functional polymers are imine-terminated polyethers such as may be obtained for example by reacting an
 45 aldehyde or ketone with a polyether polyamine, especially a polyoxypropylene diamine or triamine.

Enamine functional polymers may be prepared either from secondary amine terminated resins (i.e. polyethers) by reaction with ketones/aldehydes having one or more alpha hydrogens, or by reacting ketone/aldehyde terminated resins (bearing alpha hydrogens) with secondary amines, providing for removal of the H₂O formed in the reactions. Secondary amine terminated resins can be obtained, for example by
 50 catalytic hydrogenation of the imino-functional polymers described hereinabove. Ketone/aldehyde terminated resins may be obtained, in general, by oxidation of the corresponding secondary or primary hydroxyl terminated resin. More highly reactive enamine functional polymers can be prepared by oxidizing a primary hydroxy functional resin to the corresponding polycarboxylic acid, conversion of the said groups to orthoesters, and treatment of the latter, with an excess of a secondary amine. Each orthoester must contain
 55 at least one alpha hydrogen atom.

Polymers containing a plurality of isocyanate-reactive groups for inclusion in the isocyanate-reactive compositions of the invention preferably have molecular weights in the range from 2000 to 8000 and have isocyanate-reactive functionalities of from 2 to 4, especially 2 or 3.

Chain extenders having molecular weights below 1500 which may be present in the isocyanate-reactive compositions of the invention have been fully described in the prior art and include polyols and polyamines, especially diols and diamines. Mixtures of different chain extenders may be used if desired. Preferred chain extenders have molecular weights below 800 and often below 500.

5 Polyols which may be used as chain extenders include ethylene glycol and 1,4-butanediol.

Polyamines which may be used as chain extenders include aliphatic polyamines, especially diamines, and aromatic polyamines, especially sterically hindered diamines. Ethylene diamine and low molecular weight polyether diamines are examples of suitable aliphatic polyamines whilst suitable aromatic diamines include 3,5-diethyl-2,4-toluene diamine, 3,5-diethyl-2,6-toluene diamine and mixtures thereof, (referred to as
10 DETDA) 4,4'- and 2,4'-diaminodiphenylmethanes and mixtures thereof, 2,4- and 2,6-diaminotoluenes and mixtures thereof, 1,3,5-triisopropyl-2,4-diaminobenzene, 1,3,5-triethyl-2,4-diamino benzene 3,3',5,5'-tetraisopropyl-4,4'-diaminodiphenylmethane and the like.

Other chain extenders which may be used include imino-functional compounds as described in US Patent No. 4,794,129 and enamines.

15 The relative proportions of isocyanate-reactive polymer and chain extender present in the isocyanate-reactive compositions of the invention generally conform to the teachings of the prior art. The concentration of internal mould release composition in the isocyanate-reactive composition is typically such that the latter contains from about 0.1 to about 10 % by weight of metal salt, preferably from about 1 % to about 5 %. If
20 desired, however, a more concentrated solution or dispersion of internal mould release composition in one or more compounds containing a plurality of isocyanate-reactive groups may be prepared to act as a masterbatch to be mixed with further isocyanate-reactive compound before being used in the moulding process.

The moulding process comprises reacting a polyisocyanate composition with an isocyanate-reactive composition of the invention in a suitable mould.

25 Accordingly, in a still further aspect of the invention, there is provided a reaction system for use in making a moulded article, said system comprising the following components :

(1) a polyisocyanate composition, and

(2) an isocyanate-reactive composition comprising at least one compound having a plurality of isocyanate-reactive groups having dissolved or dispersed therein an effective amount of an internal mould
30 release composition comprising :

(a) a metal salt of an organic acid, said metal salt having mould release properties, and

(b) a compatibilising amount of a guanidine or isourea compound,

the metal salt being incompatible with the compound having a plurality of isocyanate-reactive groups in the absence of the compatibiliser, under the conditions used for preparing and/or processing said isocyanate
35 reactive composition.

The invention also provides a reaction system for use in making a reaction injection moulded article, said system comprising the following components :

(1) a polyisocyanate composition, and

(2) an isocyanate-reactive composition comprising :

40 (i) at least one polymer containing a plurality of isocyanate-reactive groups and having a number average molecular weight of from 1500 to about 12000;

(ii) at least one chain extender having a molecular weight less than 1500, and

(iii) an effective amount of an internal mould release composition comprising :

(a) a metal salt of an organic acid, said metal salt having mould release properties, and

45 b) a compatibilising amount of a guanidine or isourea compound,

the metal salt being incompatible with the combination of (i) and (ii) in the absence of the compatibiliser, under the conditions used for preparing and/or processing said isocyanate reactive composition.

Polyisocyanate compositions which may be reacted with the isocyanate-reactive compositions of the
50 invention in the moulding process may include any of the aliphatic, cycloaliphatic, araliphatic or aromatic polyisocyanates known in polyurethane or polurea chemistry, especially those that are liquid at room temperature.

Examples of suitable polyisocyanates include 1,6-hexamethylene diisocyanate, isophorone diisocyanate, cyclohexane-1,4-diisocyanate, 4,4'-dicyclo-hexylmethane diisocyanate, 1,4-xylylene diisocyanate, 1,4-
55 phenylene diisocyanate, 2,4-toluene diisocyanate, 2,6-toluene diisocyanate, 4,4'-diphenylmethane diisocyanate, 2,4'-diphenylmethane diisocyanate, polymethylene polyphenylene polyisocyanates (crude MDI) and 1,5-naphthylene diisocyanate. Mixtures of polyisocyanates can be used and also polyisocyanates which have been modified by the introduction of urethane, allophanate, urea, biuret, carbodiimide,

uretonimine or isocyanurate residues.

In general, the aromatic polyisocyanates are preferred, especially the available MDI isomers, that is to say 4,4'-diphenylmethane diisocyanate, 2,4'-diphenylmethane diisocyanate and mixtures thereof. MDI variants such as uretonimine-modified MDI and MDI prepolymers are also of great value in the moulding process.

The reaction systems of the invention may also contain other conventional ingredients of such systems, said ingredients usually being present in the isocyanate-reactive compositions of the invention. Such ingredients include catalysts, for example tin compounds and tertiary amines, surface-active agents and foam stabilisers, for example siloxane-oxyalkylene copolymers, blowing agents, for example water and low boiling halogenated hydrocarbons, flame retardants, plasticisers, dyes, fillers and reinforcing agents.

In the moulding process, the techniques and equipment used may be those that have been fully described in the prior art. RIM processes which may be used include structural (including mat-reinforced) RIM, amine extended RIM processes and "spray" RIM processes.

The invention is illustrated but not limited by the following Examples :

Example 1

A hazy solid internal mold release composition containing 20 % by weight of mold release agent was prepared by mixing at 80 ° C and cooling, a mixture of 80 g of N,N',N''-tricyclohexylguanidine and 20 g of Zinc Stearate.

A clear isocyanate-reactive composition suitable for RIM-processes was obtained by mixing at room temperature :

10 g of the above described internal mold release composition;
80 g of a high molecular weight ethylene oxide tipped triol,
having a hydroxyl value of 28 mg KOH/g, commercialized by ICI under the trade mark DALTOCEL F 2805,
20 g of DETDA.

The tricyclohexylguanidine was prepared as follows :
196.4 g of cyclohexylamine and 412.7 g of dicyclohexylcarbodiimide were charged to a 1 liter flask fitted with a mechanical stirrer and nitrogen sparge tube. The stirred reaction mixture was warmed to 180 ° C with a gentle flow of dry nitrogen. The process of the reaction was monitored by infrared spectroscopy. The consumption of the dicyclohexylcarbodiimide was followed by observing a decrease in the intensity of the absorption at 2130 cm⁻¹. After 15 hours, when no further decrease in this signal was observed, the reaction mixture was cooled and discharged to give a white crystalline solid with melting point of approx. 80 ° C.

Example 2

a) A hazy solid internal mould release composition containing 50 % by weight of mould release agent was prepared by mixing at room temperature, a mixture of 20 g of tetramethylguanidine and 20 g of Zinc Stearate

b) A clear isocyanate reactive composition suitable for RIM-processes was obtained by mixing at 80 ° C and then cooling to room temperature.
4 g of the above described internal mould release composition
80 g of DALTOCEL F2805
20 g of DETDA

Example 3

a) An internal mould release composition consisting of a hazy solution containing 33 % by weight of mould release agent was prepared by mixing at room temperature
20 g of tetramethylguanidine
10 g of Zinc Stearate

b) A clear isocyanate reactive composition suitable for RIM-processes was obtained by mixing at room temperature

6 g of the above described internal mould release composition

80 g of DALTOCEL F2805

5 20 g of DETDA

Example 4

10 A clear isocyanate reactive composition containing a mould release agent suitable for RIM processes was obtained by mixing at 80 °C and cooling to room temperature, a mixture of

4 g of diphenylguanidine

2 g of Zinc Stearate

75.2 g of DALTOCEL F2805

15 18.8 g of DETDA

Example 5 - 10

20 A clear isocyanate reactive composition containing a mould release agent suitable for RIM processes was obtained by mixing at 140 °C and cooling to room temperature, a mixture of

186 g of DALTOCEL F2805

46.5 g of DETDA

2.5 g of Zinc Stearate

25 7.5 g of a guanidine selected from :

N,N'-Di-tolylguanidine

(Dichlorophenyl)guanidine

(4-chloro-2-methylphenyl)guanidine

(4-chlorophenyl)guanidine

30 N,N'-Dixylylguanidine

(3-chlorophenyl)guanidine

The clear solutions formed were stable for at least 2 days under ambient conditions.

35

Example 11

A "B" component was prepared by mixing together 50.56 parts of a high molecular weight ethyleneoxide tipped triol having an hydroxyl value of 28 mg KOH/g commercialised by Union Carbide under the trade mark Niox E 474, 12.75 parts of DETDA and 2.68 parts of the internal mould release composition described in Example 2 a).

An elastomer was prepared by combining 35.35 parts of an "A" component consisting of an isocyanate composition containing 4,4'-diphenylmethane diisocyanate and having an NCO content of 23.0 % with 65.95 parts of the "B" component described above at a mixing ratio A/B of 0.536 on an Admiral 2000 HP RIM machine at a mould temperature of 70 °C. When filling a mould having a complex shape 17 releases were obtained with a single application of an external mould release agent. That is 17 mouldings could be easily removed from the mould without any sticking of the part to the mold or tearing of the part itself. In addition, the parts produced under these conditions showed good "green strength" and a minimum of flow lines.

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Example 12

A clear isocyanate reactive composition containing a mould release agent suitable for RIM processes was obtained by mixing at 140 °C and cooling to room temperature, a mixture of

55 80 g DALTOCEL F2805

20 g DETDA

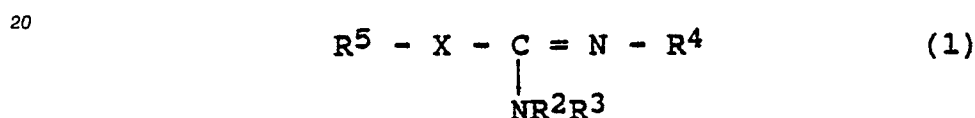
6 g N,N'-dicyclohexyl-O-ethylisourea

1 g Zinc Stearate

N,N'-dicyclohexyl-O-ethylisourea was obtained by reacting 100 g of ethanol, 20 g of dicyclohexylcarbodiimide (DCC) and 0.25 g of Cu(I)Cl as catalyst. The reactants were charged to a round bottomed flask fitted with a magnetic stirrer and reflux condensor. The reaction mixture was refluxed for four hours. The process of the reaction was monitored by IR analysis by means of the reduction in intensity of an absorption at 2130 cm⁻¹ which indicated the consumption of DCC, and the growth of a peak at 1660 cm⁻¹ which indicated formation of the isourea. When the reaction was considered to be complete the reaction mixture was cooled and excess ethanol was removed in vacuo. The crude reaction product was partitioned between water (100 ml) and hexane (100 ml). The organic layer was separated and the solvent was removed in vacuo to give the isourea product.

Claims

1. An internal mould release composition comprising :
 - (a) a metal salt of an organic acid, said metal salt having mould release properties, and
 - (b) a compatibilising amount of a guanidine or isourea compound of formula (1)



wherein X represents O, S or NR¹ and
 wherein each of R¹ of R⁵ independently or together represent, H or an organic radical and having a molecular weight below 1500.

2. A composition according to claim 1 wherein the organic radical is selected from the following :
 - a) alkyl radicals
 - b) cyclo alkyl radicals
 - c) aralkyl radicals and aryl radicals
 - d) polymeric chains containing hetero atoms, such as polyether chains, polyester chains, polycarbonates chains, polyacetal chains and the like.
3. A composition according to claim 2 wherein
 - a) the alkyl radical contains from 1 to 10 C atoms
 - b) the cycloalkyl radical contains from 5 to 7 C atoms
 - c) the aralkyl radical and aryl radical contains from 6 to 18 C atoms
4. A composition according to anyone of claim 1 to claim 3 wherein the guanidine or isourea compound has at least two of three substituents attached to the >C = N -imino-unit, joined together to form at least one non-aromatic ring.
5. A composition according to anyone of claims 1 to 4 wherein the guanidine or isourea compound of formula (I) contains from 1 to 4 guanidine or isourea groups.
6. A composition according to anyone of the preceding claims wherein the guanidine compound is diphenylguanidine.
7. A composition according to claim 5 wherein the salt is Zinc Stearate.
8. An isocyanate-reactive composition comprising at least one compound having a plurality of isocyanate-reactive groups having dissolved or dispersed therein an effective amount of an internal mould release composition according to any preceding claim, the metal salt in the mould release composition being incompatible with the compound having a plurality of isocyanate-reactive groups in the absence of the compatibiliser, under the conditions used for preparation, storage, or processing of said isocyanate reactive composition.
9. An isocyanate-reactive composition according to claim 17 containing :
 - (i) at least one polymer containing a plurality of isocyanate-reactive groups and having a number average molecular weight of from 1500 to about 12000, and

(ii) at least one chain extender having a molecular weight less than 1500,
the metal salt in the mould release composition being incompatible with the combination of (i) and (ii) in the
absence of the compatibiliser, under the conditions used for preparation, storage, or processing of said
isocyanate reactive composition.

5 10. An isocyanate-reactive composition according to claim 9 wherein the chain extender comprises an
aromatic diamine.

11. A reaction system for use in making a moulded article, said system comprising :

(1) a polyisocyanate composition, and

(2) an isocyanate-reactive composition according to any of claims 9 and 10.

10 12. A reaction system according to claim 11 wherein the polyisocyanate composition comprises at least
one diphenylmethane diisocyanate isomer and/or an MDI variant.

13. A moulded article obtained from a reaction system according to any of claims 11 and 12.

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